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L1 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN
ACCESSION NUMBER: 1992:214130 CAPLUS
DOCUMENT NUMBER: 116:214130
ENTRY DATE: Entered STN: 31 May 1992
TITLE: process for the preparation of dichlorobenzene
(1,4-dichlorobenzene) by chlorination of benzene or
chlorobenzene in the presence of Friedel-Crafts
catalysts and N-[(perfluoroalkyl)carbonyl]phenothiazin
es
INVENTOR(S): Mais, Franz Josef; Fiege, Helmut
PATENT ASSIGNEE(S): Bayer A.-G., Germany
SOURCE: Eur. Pat. Appl., 6 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
INT. PATENT CLASSIF.:
MAIN: C07C025-08
SECONDARY: C07C017-12
CLASSIFICATION: 25-3 (Benzene, Its Derivatives, and Condensed
Benzenoid Compounds)
Section cross-reference(s): 35
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 474074	A1	19920311	EP 1991-114226	19910824 <--
EP 474074	B1	19940608		
R: BE, CH, DE, FR, GB, IT, LI				
DE 4028269	A1	19920312	DE 1990-4028269	19900906
JP 04305544	A2	19921028	JP 1991-244079	19910830
JP 2902171	B2	19990607		
PRIORITY APPLN. INFO.:			DE 1990-4028269	A 19900906

PATENT CLASSIFICATION CODES:

PATENT NO.	CLASS	PATENT FAMILY CLASSIFICATION CODES
EP 474074	ICM	C07C025-08
	ICS	C07C017-12
	IPCI	C07C0025-08 [ICM,5]; C07C0025-00 [ICM,5,C*]; C07C0017-12 [ICS,5]; C07C0017-00 [ICS,5,C*]
	IPCR	C07C0017-00 [I,C*]; C07C0017-12 [I,A]
DE 4028269	IPCI	C07C0025-08 [ICM,5]; C07C0025-00 [ICM,5,C*]; C07C0017-12 [ICS,5]; C07C0017-00 [ICS,5,C*]; C07D0279-22 [ICS,5]; C07D0279-00 [ICS,5,C*]; B01J0027-128 [ICA,5]; B01J0027-06 [ICA,5,C*]; B01J0031-02 [ICA,5]
	IPCR	C07C0017-00 [I,C*]; C07C0017-12 [I,A]
JP 04305544	IPCI	C07C0025-08 [ICM,5]; C07C0025-00 [ICM,5,C*]; B01J0031-02 [ICS,5]; B01J0031-30 [ICS,5]; B01J0031-26 [ICS,5,C*]; C07C0017-12 [ICS,5]; C07C0017-00 [ICS,5,C*]; C07B0061-00 [ICA,5]

OTHER SOURCE(S): CASREACT 116:214130; MARPAT 116:214130

ABSTRACT:

A process for the preparation of dichlorobenzene comprises the chlorination of benzene or chlorobenzene in the presence of Friedel-Crafts catalysts and N-[(perfluoroalkyl)carbonyl]phenothiazines; dichlorobenzene thus prepared contains 1,4-dichlorobenzene as major fraction. Dichlorobenzene is a monomer for the preparation of polyphene sulfide and for the preparation of dyes (no data).

A reactor was charged with benzene (100 parts by weight), FeCl₃ (0.050 parts by weight), N-(trifluoroacetyl)phenothiazine (0.091 parts by weight), heated to 60° and chlorine (127 parts by weight) was passed through the mixture in 5 h. The product contained benzene (0.12% by gas chromatog.), chlorobenzene (42.90%), 1,2-dichlorobenzene (9.83%), 1,3-dichlorobenzene (0.08%), 1,4-dichlorobenzene (46.98%), and trichlorobenzenes (0.09%). The same process using N-(trichloroacetyl)phenothiazine gave a mixture containing 40.46% chlorobenzene and 40.08% 1,4-dichlorobenzene.

SUPPL. TERM: chlorination Friedel Crafts phenothiazine trifluoroacetyl benzene; regiochem Friedel Crafts fluoroacetylphenothiazine chlorination benzene

INDEX TERM: Friedel-Crafts reaction catalysts
(catalyst containing [(perfluoroalkyl)carbonyl]phenothiazine and, for regioselective dichlorination of benzene)

INDEX TERM: Regiochemistry
(of chlorination of benzene over iron trichloride/(trifluoroacetyl)phenothiazine)

INDEX TERM: Chlorination
(regioselective, of benzene over iron trichloride/(trifluoroacetyl)phenothiazine)

INDEX TERM: 7439-89-6, Iron, uses 7705-08-0, Iron trichloride, uses
ROLE: USES (Uses)
(catalyst containing [(perfluoroalkyl)carbonyl]phenothiazine and, for regioselective dichlorination of benzene)

INDEX TERM: 96124-86-6 100234-24-0, N-(Perfluorobutanoyl)phenothiazine
141135-78-6, N-(Perfluoropropanoyl)phenothiazine
ROLE: RCT (Reactant); RACT (Reactant or reagent)
(catalyst containing iron trichloride and, for regioselective dichlorination of benzene)

INDEX TERM: 95-50-1P, 1,2-Dichlorobenzene
ROLE: FORM (Formation, nonpreparative); PREP (Preparation)
(formation of, as byproduct in regioselective dichlorination of benzene over Friedel-Crafts catalysts and (trifluoroacetyl)phenothiazine)

INDEX TERM: 92-84-2DP, Phenothiazine, N-[(perfluoroalkyl)carbonyl] derivs.

ABSTRACT:

Ph 10H-phenothiazine-10-carboxylates or their (optionally halogenated or alkyl-, alkoxy-, nitro- or cyano-substituted) chlorinated derivs. and Lewis acids are useful as regioselective nuclear chlorination catalysts for aromatic hydrocarbons. The catalysts are advantageous in that the regioselectivity is not lowered even when the nuclear chlorination is conducted at a reaction temperature exceeding 45° and that the regioselectivity is enhanced with an increasing degree of chlorination. Thus, chlorination of chlorobenzene (I) 246 in the presence of FeCl₃ 0.47 and Ph 10H-phenothiazine-10-carboxylate 4.7 g at 50° in the dark for 7 h gave a product mixture containing I 44.92, o-dichlorobenzene 7.34, m-dichlorobenzene 0.05, p-dichlorobenzene (II) 47.66 and trichlorobenzene 0.04% at II selectivity 87%.

SUPPL. TERM: chlorination selectivity phenothiazinecarboxylate catalyst; chlorobenzene chlorination selectivity catalyst; arom hydrocarbon chlorination selectivity catalyst; regioselectivity chlorination catalyst phenothiazinecarboxylate; Lewis acid catalyst arom hydrocarbon chlorination

INDEX TERM: Aromatic hydrocarbons, reactions
ROLE: RCT (Reactant); RACT (Reactant or reagent)
(Lewis acids and phenothiazinecarboxylate compds. as regioselective systems for chlorination of)

INDEX TERM: Chlorination catalysts
(Lewis acids and phenothiazinecarboxylate compds. as regioselective systems for chlorination of aromatic hydrocarbons)

INDEX TERM: Lewis acids
ROLE: CAT (Catalyst use); USES (Uses)
(nuclear chlorination catalysts; with phenothiazinecarboxylate compds. as regioselective systems for chlorination of aromatic hydrocarbons)

INDEX TERM: Regiochemistry
(of regioselective nuclear chlorination catalyst for aromatic hydrocarbons)

INDEX TERM: 71-43-2, Benzene, reactions 100-41-4, Ethylbenzene, reactions 106-42-3, p-Xylene, reactions 108-88-3, reactions 108-90-7, Chlorobenzene, reactions
ROLE: RCT (Reactant); RACT (Reactant or reagent)
(Lewis acids and phenothiazinecarboxylate compds. as regioselective systems for chlorination of)

INDEX TERM: 106-46-7P, p-Dichlorobenzene 622-98-0P, p-Chloroethylbenzene 1124-05-6P, 2,5-Dichloro-p-xylene 29797-40-8P, Dichlorotoluene
ROLE: IMF (Industrial manufacture); PREP (Preparation)
(Lewis acids and phenothiazinecarboxylate compds. as regioselective systems for chlorination of aromatic hydrocarbons in manufacture of)

INDEX TERM: 66721-07-1P, Phenyl 10H-phenothiazine-10-carboxylate 199526-64-2P, 4-Chlorophenyl 10H-phenothiazine-10-carboxylate 199526-65-3P 199526-66-4P, 4-Methoxyphenyl 10H-phenothiazine-10-carboxylate 199526-67-5P, Phenyl 2-chloro-10H-phenothiazine-10-carboxylate 199526-68-6P 199526-69-7P 199526-70-0P 199526-71-1P 199526-72-2P 199526-73-3P 199526-74-4P 199540-47-1P
ROLE: CAT (Catalyst use); IMF (Industrial manufacture); PREP (Preparation); USES (Uses)
(nuclear chlorination catalysts; with Lewis acids as regioselective systems for chlorination of aromatic hydrocarbons)

INDEX TERM: 7446-70-0, Aluminum chloride, uses 7705-08-0, Iron chloride (FeCl₃), uses 174141-46-9, Antimony chloride
ROLE: CAT (Catalyst use); USES (Uses)

(nuclear chlorination catalysts; with
phenothiazinecarboxylate compds. as regioselective
systems for chlorination of aromatic hydrocarbons)
INDEX TERM: 92-39-7 92-84-2, 10H-Phenothiazine 98-88-4, Benzoyl
chloride 100-07-2, p-Methoxybenzoyl chloride 122-01-0,
4-Chlorobenzoyl chloride 874-60-2, 4-Methylbenzoyl
chloride
ROLE: RCT (Reactant); RACT (Reactant or reagent)
(reactant; reaction in manufacture of regioselective nuclear
chlorination catalyst for aromatic hydrocarbons)